

# Effects of calcium hydroxide on physical and sealing properties of canal sealers

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## Abstract

**Hosoya N, Kurayama H, Iino F, Arai T.** Effects of calcium hydroxide on physical and sealing properties of canal sealers. *International Endodontic Journal*, **37**, 178–184, 2004.

**Aim** To investigate whether  $\text{Ca}(\text{OH})_2$  in four different agents alters the physical properties (Exp. I) and sealing ability (Exp. II) of root canal sealers.

**Experiment** (Exp. I) Calcipex® (Nippon Sika-Yakuhin, Shimonoseki, Japan), Vitapex® (Neo-Dental, Tokyo, Japan), Calkyl® (Showa Yakuhin, Tokyo, Japan), and  $\text{Ca}(\text{OH})_2$  were used as  $\text{Ca}(\text{OH})_2$  agents. Four sealers were tested for flow, working time, setting time, and film thickness: Canals® (Showa Yakuhin), Canals®-N (Showa Yakuhin), Ketac™-Endo (Espe, Seefeld, Germany), and Sealapex® (Kerr, Romulus, MI, USA). Each  $\text{Ca}(\text{OH})_2$  agent was added to 10 vol.% of each sealer, and the mixture and controls without a  $\text{Ca}(\text{OH})_2$  agent were tested according to ISO specifications. Measurements were compared using Student's *t*-tests ( $P < 0.05$ ). (Exp. II) After removing  $\text{Ca}(\text{OH})_2$  agents applied to the root canals of 100

extracted human teeth, canals were filled with sealer. Controls were filled with each sealer without  $\text{Ca}(\text{OH})_2$  agents. Sealing ability was evaluated using distance of dye penetration from the apices. Dye penetration data were compared using analysis of variance and post hoc Newman–Keuls test ( $P < 0.05$ ).

**Results**  $\text{Ca}(\text{OH})_2$  agents influenced the physical properties of the sealers. Flow and setting time met ISO requirements, but film thickness and working time did not. Apical sealing ability of all four sealers was influenced by  $\text{Ca}(\text{OH})_2$  agents. The sealing ability of Sealapex® improved with all  $\text{Ca}(\text{OH})_2$  agents. The physical and sealing abilities varied among the other sealers.

**Conclusions** Contact with  $\text{Ca}(\text{OH})_2$  agents left on the canal wall caused considerable changes to the sealing ability of sealers.

**Keywords:** calcium hydroxide, physical property, root canal sealer, sealing property.

Received 24 February 2003; accepted 25 November 2003

## Introduction

The popularity of  $\text{Ca}(\text{OH})_2$  as an intracanal medicament is because of the fact that its alkalinity makes it an effective disinfectant (Caliskan & Sen 1996, Barbosa *et al.* 1997, Fava & Saunders 1999). It also has an extended time of action, and causes minimal tissue irritation (Leonardo *et al.* 1993, Siqueira & de Uzeda 1997). Paste-type  $\text{Ca}(\text{OH})_2$  is most often used for intracanal applications, as it does not evaporate and it displays low dentine permeability (Hosoya *et al.* 1998). However, placing the paste densely

into the root canal is important, as this extends the duration of high pH and enhances the disinfecting action (Siqueira & Lopes 1999).

Before root canal filling, the  $\text{Ca}(\text{OH})_2$  that has been applied to the root canal must be removed. However, complete removal of the paste is difficult irrespective of the method used (Lambrianidis *et al.* 1999). Enlarging the root canal to the next size has been recommended in order to remove  $\text{Ca}(\text{OH})_2$ , but this is not always successful (Porkaew *et al.* 1990). However, Porkaew *et al.* (1990) also found that a residual  $\text{Ca}(\text{OH})_2$  did not affect the apical sealing of root fillings. Significantly less apical leakage was detected in groups that received  $\text{Ca}(\text{OH})_2$  dressings compared to control groups, after the intracanal medicament was removed by irrigation and a reaming motion with files for sizes 40–70 (Holland *et al.* 1995). In addition, it

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has been reported that trace amounts of Ca(OH)<sub>2</sub> left in the root canal did not significantly affect sealing ability (Kontakiotis *et al.* 1997). In contrast, others have observed variations in apical sealing ability and attributed them to remaining Ca(OH)<sub>2</sub> (Caliskan *et al.* 1998). Such conflicting results suggest that Ca(OH)<sub>2</sub> may react with root canal filling materials, leading to variations in sealing ability.

Many additives are included in Ca(OH)<sub>2</sub> intracanal medicaments, and many different formulations are used for endodontic sealers. The combination of Ca(OH)<sub>2</sub> and sealer thus presumably affects physical properties and apical sealing ability.

The purpose of the present study was to determine the effects of various types of remnant Ca(OH)<sub>2</sub> in the root canal on the mechanical properties of root canal sealers and apical sealing ability after root canal filling.

## Materials and methods

### Experimental materials

Four types of Ca(OH)<sub>2</sub> were used as intracanal medicaments: Calcipex<sup>®</sup> (Nippon Sika-Yakuhan, Shimonoseki, Japan), Vitapex<sup>®</sup> (Neo-Dental, Tokyo, Japan), Calkyl<sup>®</sup> (Showa Yakuhan, Tokyo, Japan), and a 44% reagent grade Ca(OH)<sub>2</sub> mixture prepared with distilled water. Calcipex is water-based, while Vitapex is a premixed silicon oil-based preparation available in a filled syringe. Calkyl is sold as either liquid or powder, with the Ca(OH)<sub>2</sub> powder tempered with polyethylene glycol. Four types of root canal sealer were also used: Canals<sup>®</sup> (Showa Yakuhan), a zinc oxide eugenol material; Canals<sup>®</sup>-N (Showa Yakuhan), a noneugenol material; Ketac<sup>TM</sup>-Endo (Espe, Seefeld, Germany), a glass ionomer material; and Sealapex<sup>®</sup> (Kerr, Romulus, MI, USA), a Ca(OH)<sub>2</sub>-based sealer (Table 1).

## Experimental methods

### *Observation of remnant Ca(OH)<sub>2</sub> formulations in root canal walls*

As a preliminary experiment, the amount of applied Ca(OH)<sub>2</sub> remaining within the root canal was observed. After instrumentation of root canals in 20 extracted teeth, each Ca(OH)<sub>2</sub> preparation was applied densely into the root canals with a lentulo spiral filler, syringe, or root canal pluggers. Specimens were then kept at 37°C in 100% relative humidity for 2 weeks. After storage, Ca(OH)<sub>2</sub> was removed from the root canal using the master apical file with alternating irrigation with 15 mL of NaOCl and 10 mL of H<sub>2</sub>O<sub>2</sub>. Roots were longitudinally sectioned and Ca(OH)<sub>2</sub> residue in the section was observed under stereomicroscopy. A total of 40 sections, with two samples from each extracted tooth were obtained. Images were taken under  $\times 5$  enlargement and percentages of Ca(OH)<sub>2</sub>-coated surface area in relation to the surface area of the apical third of the canal were calculated using NIH image processing software (National Institutes of Health, MD, USA). Mean values for each agent were taken as the retention ratio on the root canal wall. Statistical comparisons between different sample conditions were performed using Student's *t*-test, with values of *P* < 0.05 considered significant.

### *Effects of Ca(OH)<sub>2</sub> formulations on physical properties of sealer*

Premix-type Ca(OH)<sub>2</sub> was used in suitable amounts. Powder and liquid types were prepared according to manufacturer's directions. Reagent Ca(OH)<sub>2</sub> was mixed with distilled water to a weight ratio of 44%. Ca(OH)<sub>2</sub> preparations were then added to root canal sealers in 10 vol.% and mixed well. The four types of Ca(OH)<sub>2</sub> and four types of root canal sealer were used to obtain 16 different sample conditions. Each sealer without

**Table 1** Details of experimental materials

	Application	Contents
Calcipex <sup>®</sup>	Provisionally root canal filling material	Ca(OH) <sub>2</sub> , barium sulfate, distilled water
Vitapex <sup>®</sup>	Provisionally root canal filling material	Ca(OH) <sub>2</sub> , Iodoform, silicon oil
Calkyl <sup>®</sup>	Medicaments for the root canal disinfection	Powder: Ca(OH) <sub>2</sub> Liquid: polyethylene glycol
Canals <sup>®</sup>	Root canal sealer	Powder: zinc oxide, barium sulfate, bismuth subcarbonate Liquid: clove oil, peanut oil
Canals <sup>®</sup> -N	Root canal sealer	Powder: zinc oxide, bismuth subcarbonate Liquid: fatty acids, propylene glycol
Ketac <sup>TM</sup> -Endo	Root canal sealer	Powder: calcium tungstate, lass powder Liquid: water, polyethylene polycarbonic acid, tartanic acid
Sealapex <sup>®</sup>	Root canal sealer	Ca(OH) <sub>2</sub> , barium sulphate, zinc oxide, titanium dioxide, zinc stearate

additional  $\text{Ca}(\text{OH})_2$  was tested as a control. Variations in flow, working time, setting time, and film thickness were then evaluated according to ISO-6876 (International Organization for Standardization Technical Committee/Subcommittee 2001). Five specimens were prepared and measured for each evaluation except working time. The same examiner performed each series of operations and measurements. Measurements were compared using Student's *t*-tests ( $P < 0.05$ ).

#### *Effects on apical sealing ability*

A total of 100 extracted maxillary anterior teeth that had been stored in physiologic saline were used. After sectioning the crown, each canal was cleaned and shaped using hand instruments up to apical size 60. After root canal irrigation with 15 mL of 5% NaOCl and 10 mL of 3%  $\text{H}_2\text{O}_2$ , canals were dried with paper points, and each formulation of  $\text{Ca}(\text{OH})_2$  was densely placed into 20 canals with a lentulo-spiral filler and finger pluggers (Roeko, Langenau, Germany), so that no extrusion from the apical foramen occurred. Orifices of root canals were sealed using Cavit® (Espe) and specimens were incubated for 2 weeks at 37°C and 100% relative humidity. After 2 weeks, a size 60 file with alternating irrigation was used to remove as much  $\text{Ca}(\text{OH})_2$  as possible from the root canal without scraping the canal wall. Alternating irrigation was then performed on the remaining  $\text{Ca}(\text{OH})_2$  using 15 mL of 5% NaOCl and 10 mL of 3%  $\text{H}_2\text{O}_2$ , with care taken to avoid extrusion from the apical foramen. Root canals were dried using paper points, after which the canals were filled with a single gutta-percha point using the four different kinds of root canal sealers. As with the tests for mechanical properties, five samples were prepared for each of the 16 sample conditions, and specimens with no application of  $\text{Ca}(\text{OH})_2$  were prepared as controls. Orifices of root canals were sealed using Cavit, and fingernail polish (REVLON® Nail Enamel; Revlon, NY, USA) was used to coat all root surfaces except the apical foramina. Samples were then immersed in India ink (Perikan AG, Hannover, Germany) and incubated at 37°C for 4 weeks. After 4 weeks, samples were removed from the ink, the fingernail polish was removed with hand instruments, and transparent root canal specimens were created using standard techniques. Generally, the test samples were placed in a screw-capped glass vial, demineralized in 5% nitric acid for 4 days (the acid was changed daily), and the vials agitated frequently. Roots were determined as completely demineralized when a small sharp needle could be placed through the thickest portion of the root without resistance. The demineralized roots were rinsed in run-

ning tap water and placed in distilled water for 6 h, changing the distilled water hourly. They were dehydrated in ascending concentrations of ethanol as follows: 80% overnight, 90% for 2 h, and 100% for 2 h. All roots were then air-dried and placed in methyl salicylate to complete the clearing process (Fulkerson *et al.* 1996). Apical microleakage from specimens was assessed by an examiner measuring the most extensive linear dye penetration using a digital caliper and a microscope accurate to 0.01 mm.

Dye penetration data were compared using analysis of variance and post hoc Newman–Keuls test ( $P < 0.05$ ).

## Results

### $\text{Ca}(\text{OH})_2$ on the root canal wall

Table 2 shows  $\text{Ca}(\text{OH})_2$  retention in the apical thirds of sections. Vitapex remained in large amounts in the apical canal wall and on the root canal wall near the orifice. Significantly more residual Vitapex was observed than the other three types of  $\text{Ca}(\text{OH})_2$  ( $P < 0.05$ ).

### Physical properties

#### *Flow*

Most samples had a tendency for reduced flow compared to controls. Many variations were observed by combining  $\text{Ca}(\text{OH})_2$  with root canal sealers. Reagent  $\text{Ca}(\text{OH})_2$  decreased flow in all sealers, with a marked decrease of 12.48 mm with Ketac-Endo to 21.70 ± 1.36 mm compared to controls. However, all variations complied within the ISO-determined diameter standards of ≥20 mm (Fig. 1).

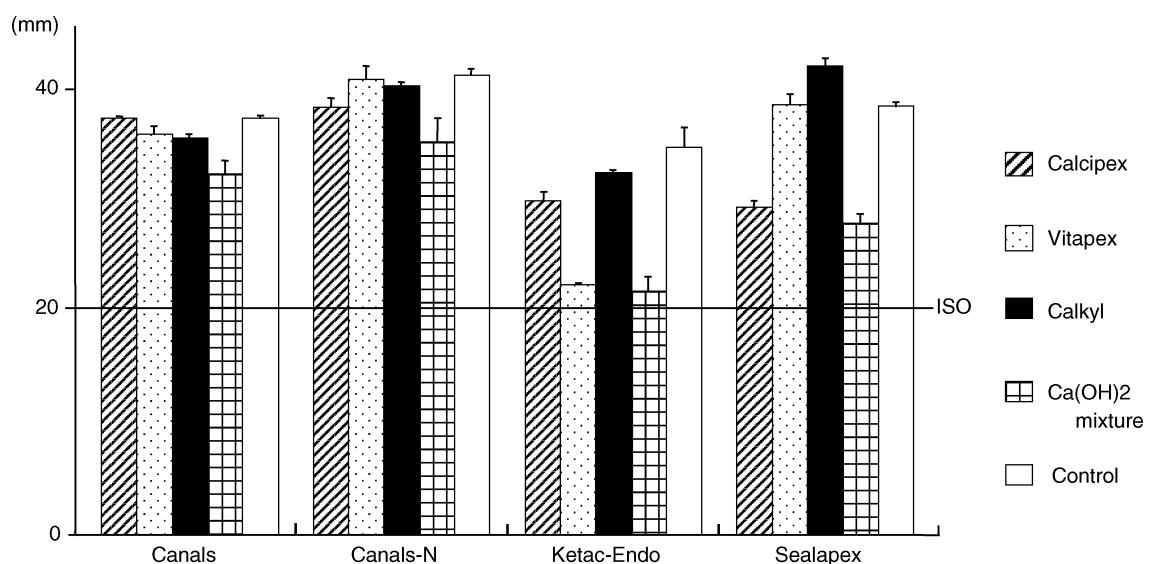
#### *Working time*

Addition of  $\text{Ca}(\text{OH})_2$  impacted substantially on setting time for all samples except the combination of Canals with Calcipex. Particularly large influences were observed for the combinations of Canals and Canals-N

**Table 2**  $\text{Ca}(\text{OH})_2$  retention expressed as ratio of surface areas of coated area to apical third of section (%;  $n = 5$ )

$\text{Ca}(\text{OH})_2$ agent	Mean ± SD
Calcipex®	27.93 ± 8.69
Vitapex®	59.39 ± 10.88*
Calkyl®	34.85 ± 11.64
Reagent $\text{Ca}(\text{OH})_2$	32.36 ± 9.84

Significantly different from other agents (\* $P < 0.05$ ).

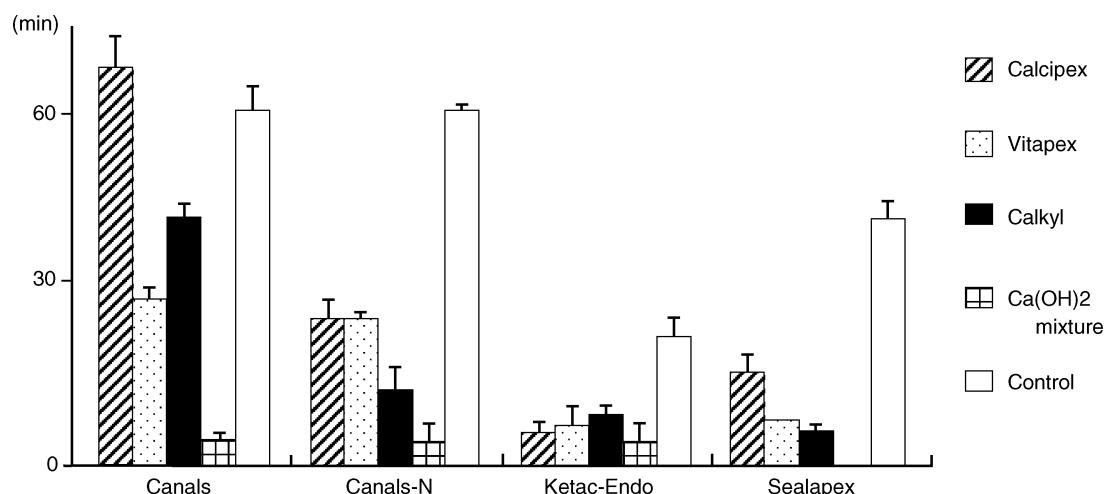


**Figure 1** Flow value under several conditions (mean  $\pm$  SD;  $n = 5$ ). ISO standard and diameter after pressuring for flow measurements. All results fit within ISO-determined diameter standards.

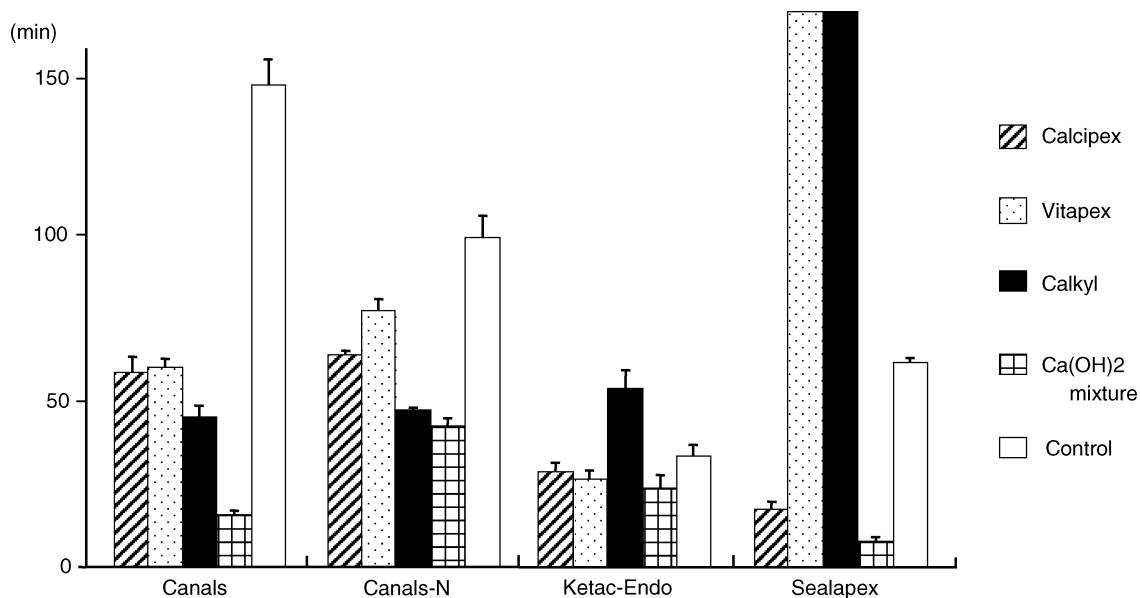
with reagent  $\text{Ca}(\text{OH})_2$ . Decreases of 53.51 min with Canals and 54.96 min with Canals-N were observed. Working time for Canals with Calcipex increased by 6.10 min, but this did not represent a significant difference. Combining Sealapex with reagent  $\text{Ca}(\text{OH})_2$  resulted in extremely short working time, and measurements were unable to be completed using ISO standard measuring methods (Fig. 2).

#### Setting time

All  $\text{Ca}(\text{OH})_2$  additives caused significant variations with Canals. Addition of  $\text{Ca}(\text{OH})_2$  caused extreme reductions in setting time for most sample conditions. Setting time for Canals with reagent  $\text{Ca}(\text{OH})_2$  was reduced by 129.58 min compared to controls. However, setting took  $\geq 150$  min for Sealapex with Vitapex or Calkyl (Fig. 3).



**Figure 2** Working time under several conditions. Sealapex was particularly affected (mean  $\pm$  SD;  $n = 5$ ). The reagent  $\text{Ca}(\text{OH})_2$  also exerted a substantial effect on all types of root canal sealers.



**Figure 3** Setting time under several conditions (mean  $\pm$  SD;  $n = 5$ ). Sealapex mixed with all kinds  $\text{Ca}(\text{OH})_2$  resulted in extremely large variations.

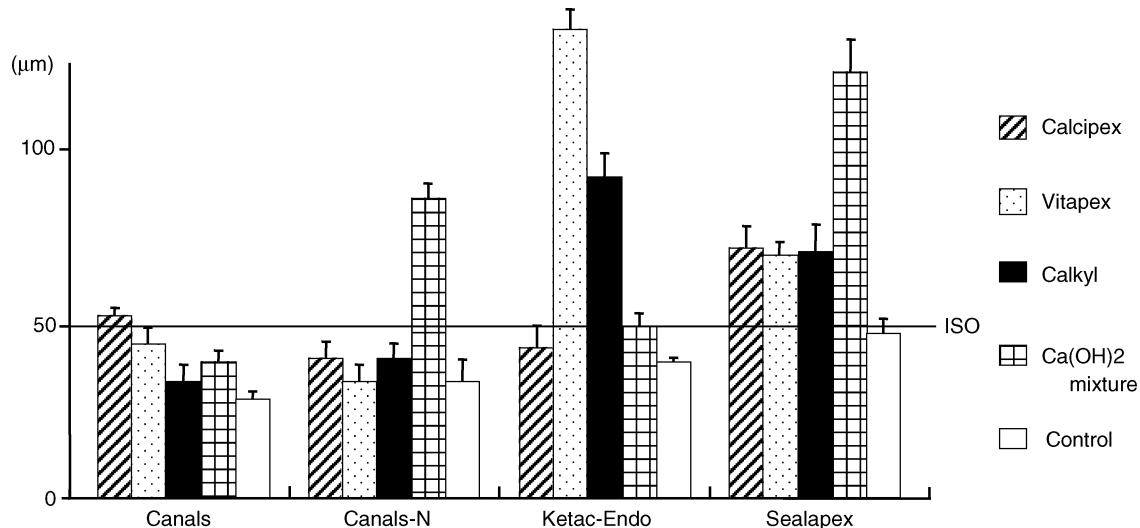
#### Film thickness

Increased film thickness was observed for most combination of materials. Combinations of Canals with Calcipex, Canals-N with reagent  $\text{Ca}(\text{OH})_2$ , and Ketac-Endo with Vitapex or Calkyl resulted in thicknesses  $> 50 \mu\text{m}$ , values that do not meet ISO standards. Particularly large effects were observed for all  $\text{Ca}(\text{OH})_2$  formula-

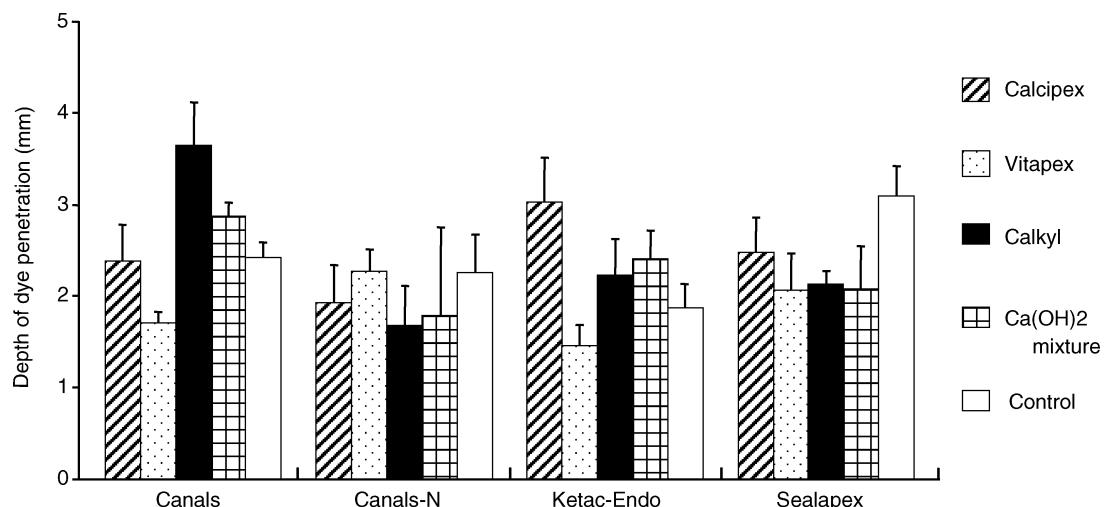
tions with Sealapex. The biggest change was seen for Ketac-Endo with Vitapex, resulting in a thickness of  $98.37 \mu\text{m}$  (Fig. 4).

#### Apical sealing ability

Marked variations were observed when combining  $\text{Ca}(\text{OH})_2$  with root canal sealers. Significantly better



**Figure 4** ISO standard and film thickness after pressuring under several conditions (mean  $\pm$  SD;  $n = 5$ ). A large increase in thickness resulting in deviation from ISO standards was observed with all  $\text{Ca}(\text{OH})_2$  preparations in combination with Sealapex.



**Figure 5** Depth of dye penetration for measuring apical sealing ability under several conditions (mean  $\pm$  SD;  $n = 5$ ). Improvement in sealing ability was observed with all  $\text{Ca}(\text{OH})_2$  preparations in combination with Sealapex.

sealing ability was achieved for Canals or Ketac-Endo with Vitapex (1.68 and 1.43 mm, respectively), and Canals-N with Calkyl (1.64 mm) than for other combinations. However, Canals with Calkyl or reagent  $\text{Ca}(\text{OH})_2$ , and Ketac-Endo with Calcipex, Calkyl, or reagent  $\text{Ca}(\text{OH})_2$  led to reduced sealing ability (Fig. 5).

## Discussion

Removing all the  $\text{Ca}(\text{OH})_2$  that had been placed in the root canal was impossible using a file and alternating irrigation. The water-based Calcipex was easy to handle, and was the most easily removed, but great care had to be taken to avoid extrusion via the apical foramen. At the other end of the spectrum, silicon oil-based Vitapex was sticky and adhered to large areas of root canal wall, resulting in significant amounts remaining after cleaning. In any case, removal of all  $\text{Ca}(\text{OH})_2$  from the root canal could not be achieved for any conditions tested.

ISO standards for mechanical properties of root canal sealers usually involve five tests. However, testing of material solubility and disintegration was not undertaken in this study. Some combinations of root canal sealers and  $\text{Ca}(\text{OH})_2$  agents, although setting enough to be measured with ISO hardening time measurements, demonstrated such brittle and rough surfaces that preparing the disc samples for measuring solubility and disintegration was difficult.

Extreme reduction in flow and working time results in an inability to work effectively with a material, increasing the chances of a void being created. In addition,

decreased film thickness is believed to make sufficient condensation of the root canal filling difficult to achieve.

Variations in sealing ability may be caused by chemical reactions or interactions between types of  $\text{Ca}(\text{OH})_2$  and root canal sealer, but the details of these interactions remain unknown. The improvement of sealing observed in this study cannot be directly interpreted as a clinically relevant result, as testing was performed *in vitro* and did not mirror a clinical scenario.

The results of this study confirm those of previous reports (Porkae *et al.* 1990, Holland *et al.* 1995, Kontakiotis *et al.* 1997), which found that  $\text{Ca}(\text{OH})_2$  remaining inside root canals improves apical sealing ability. However, these results are based on measurements conducted over a short period of time using laboratory samples. Rapid setting or remnant  $\text{Ca}(\text{OH})_2$  with an oil-based sealer may indeed prevent dye penetration, but technical errors occur readily and sealing ability may be impaired with time. As root canal sealers with added  $\text{Ca}(\text{OH})_2$  resulted in extremely rough surfaces, more detailed long-term clinical surveys are required. Furthermore, the existence of smear layer influences apical leakage. Removal of the smear layer reportedly results in significantly reduced microleakage in teeth obturated using AH 26 sealer (Economides *et al.* 1999). In this study, the smear layer was not performed. Had this been performed the results may have been different. After  $\text{Ca}(\text{OH})_2$  dressing, use of gutta-percha points combined with  $\text{Ca}(\text{OH})_2$  for root canal filling exhibited significantly less apical leakage than gutta-percha points alone (Holland *et al.* 1996).

Finally, as the exact amount of Ca(OH)<sub>2</sub> left in the root canal was not investigated, differing amounts may exert different degrees or reverse effects on sealing ability.

It has been reported previously that trace amounts of water or exudate remaining in the root canal during root canal filling can affect apical sealing ability (Hosoya *et al.* 2000). In other words, the water included in residual Ca(OH)<sub>2</sub> may affect mechanical properties and apical sealing ability. Ca(OH)<sub>2</sub> should therefore be removed as much as possible during root canal filling, and consideration should be given to Ca(OH)<sub>2</sub> remaining in the root canal when selecting a root canal sealer.

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